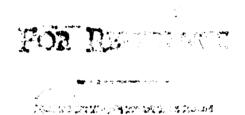


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EFFECTS OF YTTRIUM, ALUMINUM, AND CHROMIUM CONCENTRATIONS IN BOND COATINGS ON THE PERFORMANCE OF ZIRCONIA-YTTRIA THERMAL BARRIERS



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#### SUMMARY

The effects of yttrium concentration in NiAlY and NiCrY and of chromium and aluminum concentrations in NiCrAlY bond coatings on the cyclic furnace life of thermal barrier coatings were studied. Also, the effect of bond coating thickness on life was studied. The evaluations were done in cyclic furnace tests between  $990^{\rm o}\text{-}280^{\rm o}$  and  $1095^{\rm o}\text{-}280^{\rm o}$  C on solid specimens.

On the basis of the data obtained in this study, it was established that the presence and the concentration of yttrium in the bond coating is very critical. Without yttrium in the Ni-Cr, Ni-Al, and Ni-Cr-Al bond coatings, the thermal barrier systems fail very rapidly. The optimization of yttrium, and to a lesser extent, chromium and aluminum concentrations in the nickel-base alloy bond coatings leads to a very significant improvement of the thermal barrier systems.

The data indicates that the best bond coating of those studied was Ni-25.7Cr-5.6Al-0.32Y followed closely by Ni-19.9Cr-19.2Al-0.33Y and Ni-19.3Al-0.52Y. When these bond coatings were coupled with  $\rm Zr0_2-7.8Y_20_3$ , the oxide withstood 1500, 1500, and 1389 1-hour cycles without failures between 990°-280° C and 246, 208, and 198 1-hour cycles between  $\rm 1095^{\circ}-280^{\circ}$  C, respectively. (Compositions are reported in weight percent).

Furthermore, the data suggest that the thickness of the bond coating has a very significant effect on the life of the thermal barrier system. By increasing the thickness of the bond coating in the Ni-16.4Cr-5.8Al-0.32Y/ZrO<sub>2</sub>-7.8Y<sub>2</sub>O<sub>3</sub> system from 0.003 to 0.012 cm, the cyclic life between 990°-280° C increased from 1419 1-hour cycles to greater than 1610 1-hour cycles with no failure. By increasing the thickness from 0.005 to 0.013 cm, the life increased from 46 to 104 1-hour cycles between  $1095^{\circ}$ -280° C. By further increasing the bond coating thickness in the improved Ni-25.7Cr-5.6Al-0.32Y/ZrO<sub>2</sub>-7.8Y<sub>2</sub>O<sub>3</sub> thermal barrier system from about 0.011 to 0.020 cm, the life increased from 246 to 646 1-hour cycles between  $1095^{\circ}$ -280° C.

## INTRODUCTION

In the past, attempts were made to use thermal barrier coating systems in regeneratively cooled rocket engines (refs. 1 to 3), on aircraft gas engine components (refs. 4 and 5), and on air-cooled turbine blades and vanes (ref. 6). The overall success of all of these past attempts was rather marginal. Nipjes (ref. 7) evaluated various bond coatings coated with calcia-stabilized zirconia. He reported that Nimonic 115 alloy coated with 1 mm zirconia layer had good thermal insulation properties and thermal shock resistance without loss of mechanical strength of the substrate.

Recently, significant progress has been made in improving the performance and the adherence of the two-layer thermal barrier system to metallic substrates (refs. 8 to 11). The data reported in these references indicate that a NiCrAlY/Zr02-Y203 duplex system is better than NiCrAlY/Zr02-MgO or NiCrAlY/Zr02-

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CaO system and that sertain specific NiCrAl-Y alloy bond coating compositions and  $Zr0_2-Y_20_3$  barrier oxide compositions can withstand 2000 1-hour cycles in Mach 1.0 burner rig at 1470° C surface temperature.

The performance and the adherence of such NiCrAlY/ZrO $_2$ -Y $_2$ O $_3$  thermal barrier system is very sensitive to the yttrium concentration in the bond coating and to the yttria concentration in the zirconium dioxide thermal barrier layer(ref. 11). The best two-layer thermal barrier systems reported in reference 11 were Ni-16.4Cr-5.1Al-0.15Y and Ni-17.0Cr-5.4Al-0.35Y bond coatings with ZrO $_2$ -6.2Y $_2$ O $_3$  and ZrO $_2$ -7.8Y $_2$ O $_3$  (all in wt %) stabilized zirconium dioxide thermal barrier layers.

In reference 11, however, no mention is made of how the adherence and the performance of the NiCrAlY/ $Zr0_2$ - $Y_20_3$  thermal barrier system would be affected when no yttrium, or no aluminum, or no chromium is present in the bond coating or when the concentrations of chromium and aluminum are changed. Furthermore, the data in reference 11 indicate that the performance of the thermal barrier system is very sensitive to temperature in an oxidizing atmosphere and that the thin coating is gradually degraded by oxidation. Therefore, it is also of interest to know what effect the thickness of the bond coating has on the adherence and the performance of the thermal barrier system.

To further improve the NiCrAlY/ZrO $_2$ -Y $_2$ O $_3$  thermal barrier system for aerospace applications, this study was conducted to examine the effects of yttrium concentration in the bond coating, the effects of aluminum and chromium concentrations in the bond coating, as well as the effect of the thickness of the bond coating on the life and the adherence of the two-layer thermal barrier systems. The evaluations were done in a cyclic furnace. The coated specimens were heated to either about  $990^{\circ}$  or  $1095^{\circ}$  C in 6 minutes, held at temperature for 60 minutes, and cooled to about  $280^{\circ}$  C in 60 minutes.

# MATERIALS, APPARATUS, AND PROCEDURE

#### Materials

The compositions of plasma spray powders of Ni-Cr-Al-Y (-200 to  $\pm$ 325 mesh) and yttria-stabilized zirconia (-200 to  $\pm$ 325 mesh) are presented in table I. The bond and oxide coating compositions throughout this report are expressed in weight percent. The substrates were the conventionally cast nickel-base alloys - B-1900 plus hafnium (Hf), and MAR-M-200 plus hafnium and the results of the impurity analyses are also reported in table I. Flat specimens, 2.5x1.3x0.25 cm, with all corners and edges rounded to about 0.16 cm radius were used.

## Apparatus and Procedure

<u>Plasma spray coating deposition.</u> - All specimen surfaces were grit blast cleaned with alumina and within 10 minutes the NiCrAly, NiAly, or NiCry bond coating was applied with a plasma spray gun(350 amperes and 28 volts) to a nominal thickness

of about 0.010cm. Bond coat and oxide thicknesses were measured with a vernier caliper. Values reported are maximums. However, the bond coating thicknesses on some of the specimens were also checked at various locations on the samples using a micrometer having a 0.65 cm shaft. These measurements showed that the bond coating thickness on any specimen varied by  $\pm 0.004$  cm. In cases where the effect of bond coating thickness on the performance of the thermal barrier system was studied, the thickness ranges as determined with a micrometer are reported in the tabular presentations of these data. The  $\text{ZrO}_2\text{-Y}_2\text{O}_3$  coatings were applied by a plasma spray gun (550 amperes and 33 volts) within 20 minutes of completing the bond coating. Zirconia coatings were nominally 0.038 cm thick and the variation in oxide coating thickness was about  $\pm 0.005$  cm as determined with a micrometer at various locations on the specimen.

Plasma spraying of both coatings was done in an open air environment using argon as the plasma gas. The plasma spray gun-to-substrate distance was maintained at between 13 and 15 cms.

Coating tests - In the furnace cyclic tests, coated specimens were heated in air to  $990^\circ$  or  $1095^\circ$  C. The cycle in all cases consisted of 6 minutes heatup, 60 minutes at temperature, and 60 minutes cooling to about  $280^\circ$  C. Specimens from all of these tests were normally removed from the furnace and inspected visually about every 12 cycles until a visible external crack appeared in the oxide coating. The specimens were removed from the furnace for inspection at temperatures between  $350^\circ$  and  $400^\circ$  C. Because of this arrangement, coated specimens that withstood 1500 1-hour cycles at  $990^\circ$  C were cooled to room temperature at least 125 times. The temperature in the furnace was measured with a platinum-platinum-13 percent rhodium thermocouple and it is estimated that the reported temperature values are accurate within  $\pm 0.7$  percent.

Such cyclic furnace tests appear to be reliable for comparison purposes as indicated by the data in references 8, 9 and 11. The data in these references clearly show that the thermal barrier system having the longest life in the cyclic furnace also had the longest life in cyclic natural gas-oxygen torch burner rig and in cyclic Mach 1.0 burner rig tests.

### RESULTS AND DICUSSION

Effect of yttrium-free Ni-Cr and Ni-Al bond Coatings. - The data in table II indicate that the introduction of an yttrium-free Ni-Cr, Ni-Al, or Ni-Cr-Al bond coating between the substrate and the thermal barrier oxide layer slightly improves the life of the yttria-stabilized zirconia thermal barrier coatings compared to those with no bond coating. The appearance of an external crack in the thermal barrier oxide layer is considered to be the failure of the thermal barrier system. In the oxide coated specimens that contained no bond coating, the substrate surface was blast cleaned with high purity alumina. Therefore, the substrate surface was relatively smooth (100 rms) as compared to the surfaces of the plasma sprayed bond coating (350 rms). It is believed that the surface roughnesses of the Ni-19.8Al, Ni-20.2Cr, and Ni-16.2Cr-5.5Al bond coatings were very similar since all three bond coatings were applied under similar conditions.

The data in table II show that at 990°C the differences in the lives of the ZrO<sub>2</sub>-7.8Y<sub>2</sub>O<sub>3</sub>coatings applied directly on alumina grit blast cleaned B-1900&Hf versus the Ni-19.8Al bond coating which was plasma sprayed onto MAR-M-200&Hf is small when compared to the differences in lives between the three yttrium-free bond coatings. As the data in table II also show, similar results were obtained with ZrO2-11.5Y2O3 coatings. If it is assumed that the mechanical forces due to interlocking, play a major role in the performance of the thermal barrier system, then increasing the roughness from 100 rms on the B-1900&Hf substrate to 350 rms on the Ni-19.8Al bond coating should result in lower interfacial stresses, less cracking, and a significant increase in the life of thermal barrier system. Furthermore, the three bond coatings reported in table II should have similar lives. This is not the case as indicated by the data in table II. Therefore, it is believed that the most important role in the performance of the thermal barrier system is played by the composition of the bond coating. It is believed that better adherence of the oxide layer to the metallic bond coating is probably dependent upon oxidation resistance and some kind of chemical-electrostatic bonding. Although the conposition of the bond coating is of the primary importance in the performance life of the thermal barrier system, it may also be true that in some cases the surface roughness should at least have some influence on the adherence of the oxide layer to the bond coating and the performance life of the thermal barrier system. Indeed, it was reported in reference 12 that the bond coating must have considerable roughness in order to increase the adherence of the oxide layer. The author believes that as the surface area increases due to roughness, the chemical-electrostatic bonding forces responsible for the adherence of the oxide layer to the bond coating can act through a larger area and thus increase the adherence strength between the oxide layer and the bond coating.

Failure of the ZrO2-Y2O3 thermal barrier layer applied directly on the B-1900&Hf substrate with no bond coating occurred by interfacial oxidation, separation of the oxide from the substrate, and formation of a crack just about perpendicular to the substrate (fig. 1). The crack occurred at the highly stressed corner/edge region. Closer examination of the oxide coating revealed the presence of many small cracks in the ZrO<sub>2</sub>-7.8Y<sub>2</sub>O<sub>3</sub>and ZrO<sub>2</sub>-11.5Y<sub>2</sub>O<sub>3</sub> thermal barrier layers and these internal cracks were either parallel or at acute angles to the substrate as shown in figure 1. Both the ZrO2-7.8Y2O3 and ZrO2-11.5Y2O3 thermal barrier layers were found to contain a second dark gray phase before and after testing. It seems that this dark gray phase is present in larger quantity in ZrO2-11.5Y2O3. It is believed that this dark gray regions are yttria-stabilized zirconia that contain excess yttria (Fig. 1). Thus, the presence of the dark gray regions or the non-uniform distribution of yttria and yttria-stabilized zirconia cubic phase in the monoclinic zirconia could affect the performance life of the thermal barrier system. Therefore, the effect of this non-uniform distribution on the life of the thermal barrier system remains to be evaluated.

Effects of yttrium on NiAl and NiCr bond coats. - The data in tables III and IV show that the presence of yttrium in the Ni-Cr and Ni-Al bond coating, and in Ni-Cr-Al as shown in reference 11, has a very significant beneficial effect

on the performance of the thermal barrier system. It was found that the Ni- $20.2\text{Cr/Zr}_{02}$ -7.8 $\text{Y}_{2}_{03}$  system failed at 990° C after 205 1-hour cycles and at 1095° C after only 27 cycles, whereas, the Ni-19.8Cr-0.5Y/Zr0 $_2$ -7.8Y $_2$ 0 $_3$  system did not fail after 1550 1-hour cycles at 9900 C but failed after only 56 1-hour cycles at 10950 C. Similar results were obtained for Ni-Al and Ni-Al-Y bond coatings. Thus, the Ni-19.8Al/Zr02-7.8Y203 system failed at  $990^{\circ}$  C after 103 1-hour cycles. With a 0.52Y addition this system did not fail after 1389 1-hour cycles to 990° C and at 1095° C it only failed after 198 1-hour cycles. Based on the 1095° C data in tables III and IV, Ni-Al-Y bond coatings are more desirable than Ni-Cr-Y bond coating. Thus, the data in tables III and IV confirm that the presence of yttrium in the Ni-Cr and Ni-Al bond coatings has a strong influence on the life of the yttria-stabilized zirconia thermal barrier coating. Comparison of the data in tables III and IV at 990°C show that the substitution of chromium for aluminum or aluminum for chromium does not affect the life of yttria-stabilized zirconia thermal barrier coatings as much as does the elimination of yttrium.

The data in tables III and IV further suggest that a significant increase in either the yttrium concentration in the bond coatings or the yttria concentration in zirconia, results in a significant decrease in the life of the thermal barrier system. Thus, the Ni-19.5Cr-1.53Y/Zr0 $_2$ -7.8Y $_2$ 0 $_3$  system at 990°C failed after 300 1-hour cycles as compared to no failure after 1550 1-hour cycles for the Ni-19.8Cr-0.53Y/Zr0 $_2$ -7.8Y $_2$ 0 $_3$  system. Ni-19.4Al-1.60Y/Zr0 $_2$ -7.8Y $_2$ 0 $_3$  failed after 233 1-hour cycles and Ni-19.3Al-0.52Y/Zr0 $_2$ -7.8Y $_2$ 0 $_3$  did not fail after 1389 1-hour cycles. Similar trends were obtained when the temperature was increased to 1095° C and also when the above bond coatings were coupled with 11.5Y $_2$ 0 $_3$ -, 17.4Y $_2$ 0 $_3$ -, and 24.4Y $_2$ 0 $_3$ - stabilized zirconia thermal barrier coatings. Therefore, the data in tables III and IV confirm the trend observed in reference 11.

The metallographic studies of the tested specimens coated with Ni-Cr, Ni-Al, Ni-Cr-Y, or Ni-Al-Y bond coating and  $Zr0_2-Y_20_3$  showed some interesting results. The  $Zr0_2-7.8Y_20_3$  coated specimens having the yttrium-free Ni-20.2Cr or Ni-19.8Al bond coating failed at the substrate-bond coating interface (Figs. 2a and b). This is the first time in this author's experience that the failure occurred in this location. Both photomicrographs in figure 2 show that Ni-20.2Cr and Ni-19.8Al bond coatings were completely oxidized. Similar results were also observed for these bond coatings when coupled with higher yttria-stabilized zirconia thermal barrier coatings. These failures exemplify the fact that even with the more resistant alloys, bond coating oxidation can be a major life limiting factor.

Based on literature, it was anticipated that yttrium-free bond coatings would undergo rapid oxidation. It was further anticipated that the addition of yttrium to the nickel-base alloy bond coatings would improve the oxidation resistance and also significantly improve the performance life of the thermal

barrier system. The photomicrographs in figure 3 show that after longer exposure times Ni-19.8Cr-0.53Y bond coating has been extensively oxidized at 990°C but not to the same extent as was the Ni-20.2Cr bond coating. The Ni-19.3Al-0.52Y bond coating was somewhat less oxidized at the same temperature than was the Ni-19.8Cr-0.53Y bond coating. The photomicrographs of the specimens coated with Ni-19.8Cr-0.53Y/Zr02-7.8Y203 and Ni-19.3Al-0.52Y/Zr02-7.8Y203 systems showed that both of these systems are degraded in a similar manner. Small cracks were identified within the oxide layer as shown in figure 3 but no cracks were observed leading to the surface. Photomicrographs of these systems after exposure at 10950 C are shown in figures 4a and b. At this temperature the thermal barrier systems failed through the extension of internal cracks to the surface of the oxide layer. Surface connected cracks normally occurred at the corners or along the edges of the specimens. The internal cracks are propagated to the surface mostly at right angles to the bond coating, but some acute angle cracks were also observed. Furthermore, from the comparison of the photomicrographs in figures 3 and 4 it may be concluded that the Ni-Cr-Y and Ni-Al-Y bond coatings were not as extensively oxidized at 1095° C after 56 and 198 1-hour cycles as they were at 990° C after 1550 and 1389 1-hour cycles, respectively.

The higher magnification photomicrograph of figure 4b in figure 5 shows that the Ni-19.3Al-0.52Y bond coating has many oxide stringers. Preliminary electron microprobe data indicate that these oxide stringers are principally aluminum oxides. Higher aluminum concentration regions which probably are also aluminum oxides were also observed along the grain boundaries of the plasma deposited particles in the as-sprayed condition. These oxide layers between the bond coating particles may grow by the diffusion of oxygen. It is also possible that small cracks could have been formed along some of these grain boundaries, thus, facilitating the diffusion of oxygen.

Metallographic evaluation of the Ni-19.5Cr-1.53Y or Ni-19.4Al-1.60Y bond coating, when coupled with various yttria-stabilized zirconia thermal barrier coatings, revealed that these barrier systems failed in a manner similar to the thermal barrier systems having about 0.5 wt % yttrium in the bond coat. At the time of oxide coating failure, the NiCr-1.53Y and Ni-Al-1.60Y bond coatings did not appear to be as heavily oxidized as those with lower yttrium concentrations. However, the thermal barrier systems with higher yttrium bond coat concentrations fail much more rapidly as indicated by the data in tables III and IV.

It was also observed that as the yttrium concentration in the bond coating increased, the rate of weight gain during cyclic oxidation increased. One of the explanations for this behavior might be that yttrium in the bond coating is diffusing toward the bond coating-oxide layer interface and is gettering oxygen at this interface and also along the grain boundaries of the plasma sprayed particles. A second explanation might be that the incorporation of yttrium in the bond coating in oxide scale at the bond coating-zirconia interface and around the plasma sprayed particles increases the rate of oxygen diffusion in the scale. Preliminary electron microprobe data suggest that yttrium is diffusing toward the bond coating-oxide coating interface. More extensive electron microprobe and scanning electron microscopy evaluations must be done in order to acquire a

more comprehensive understanding of this aspect of the complex bond coating degradation process.

Effect of bond coat Cr and Al content. – The data in table V show that the chromium and aluminum concentrations in the bond coating also have a significant effect on the performance of the thermal barrier system. The various nickel-base alloys containing between 16.4 to 25.7Cr, 5.8 to 10.6Al, and about 0.32Y, were coupled with Zr02-7.8Y203 and Zr02-11.5Y203. These thermal barrier systems withstood 1500 1-hour cycles to 990° C without failures. At 1095° C, the results from the cyclic furnace tests suggest that the best thermal barrier system was the one with Ni-25.7Cr-5.6Al-0.32Y bond coating. This bond coating, when coupled with the Zr02-7.8Y2033thermal barrier coating, failed after 246 1-hour cycles as compared to the Ni-16.4Cr-5.8Al-0.32Y/Zr02-7.8Y203 system which failed after only 114 1-hour cycles. Furthermore, the comparison of the data in table V and in reference 11 shows that the Ni-25.7Cr-5.6Al-0.32Y bond coating coupled with zirconia stabilized with 7.8, 11.8, or 17.4Y203 gives longer lives than Ni-16.4Cr-5.1Al-0.15Y, when coupled with these oxides (refs. 11).

Indeed the cyclic furnace test results in table V suggest that the Ni-Cr-Al-Y bond coating containing about 16Cr and 6Al does not seem to perform as well in this test as do the bond coatings containing higher Cr and/or Al levels. The exception seems to be the Ni-Al-Y and Ni-Cr-Y bond coatings as the data in tables III and IV suggest. The data in table IV show that Ni-19.3Al-0.52Y coupled with  $Zr0_2$ -7.8Y $_20_3$  withstood 1389 1-hour cycles and no failure between 990° C and about 280° C and 198 1-hour cycles between 1095° C and about 280° C and failure. It is possible that this bond coating could further be optimized by changing the concentrations of aluminum and yttrium. The data in table III show that Ni-19.8Cr-0.53Y bond coating coupled with  $Zr0_2$ -7.8Y $_20_3$  withstood 1550 1-hour cycles and no failures between 990° and about 280° C but only 56 1-hour cycles between 1095° and about 280° C.

As already shown, the yttrium, chromium, and aluminum concentrations in the bond coating have a very significant influence on the life of the thermal barrier system. Thus, on the basis of the cyclic furnace data reported in tables IV and V and the data reported in reference 11 it may be concluded that for yttria-stabilized zirconia thermal barrier coatings to have long lives, the bond coating probably should contain more than 20 weight percent chromium, more than 5 weight percent of aluminum, and probably less than 0.3 weight percent yttrium. It is believed that such bond coating compositions will have high resistance to degradation through oxidation.

The photomicrograph in figure 6 shows that the Ni-25.7Cr-5.6Al-0.32Y bond coating is representative of all those other bond coatings discussed in table V before testing. The photomicrograph shows that the Ni-25.7Cr-5.6Al-0.32Y bond coating contains some oxide particles and oxide stringers at particle boundaries, iis quite rough and convoluted, and is not of uniform thickness. The large dark areas within the oxide coating were probably caused by pull-out of particles during cutting and polishing of the specimens. The comparison of the photomicrographs in figures 6, 7, 8, and 9 indicate that the degradation of the bond

coating through oxidation increases from the Ni-25.7Cr-5.6Al-0.32Y, to Ni-19.9Cr-19.2Al-0.33Y, to Ni-16.6Cr-10.6Al-0.33Y bond coating. Furthermore, the degradation of the bond coating appears to be greater at 1095° than at 990° C. Thus, the degree of degradation of the bond coating, or the resistance of the bond coating to oxidation seems to be directly related to the life of the thermal barrier system. As the oxidation resistance of the bond coating increases, the life of the thermal barrier system increases. The oxidation resistance order established from the metallographic evaluations of the various bond coatings reported in table V and whose photomicrographs are shown in figures 6 to 9 seems to be supported by the data presented in reference 13. Barrett and Lowell (ref. 13) reported that the Ni-31Cr-11Al and Ni-27.3Al alloys are more oxidation resistant than any other Ni-Cr-Al composition studied.

All of the thermal barrier systems consisting of the bond coating reported in table V and yttria-stabilized zirconia failed in a manner similar to the systems consisting of the Ni-Al-Y or Ni-Cr-Y bond coating and yttria-stabilized thermal barrier coating. Specimens tested at 990°C did not fail even after 1500 1-hour cycles and no external cracks in the thermal barrier oxide coatings were observed. Metallographic evaluations showed that the oxide coating did contain microcracks. All of the specimens tested at 1095°C failed by forming an external crack at the corner or along the edge of the coated specimen. These specimens also contained microcracks within the oxide coating. Consequently, from the data reported in this study and the data reported in references 8, 9, and 11, it may be concluded that whenever the bond coating contains yttrium the failure seems to originate within the oxide coating near the bond coating-thermal barrier oxide coating interface. If no yttrium is present in the bond coating, then it seems that the failure is originated at the substrate-bond coating interface.

Effect of bond coating thickness. - The data in table VI show that the thicker the bond coating, the longer the life of the thermal barrier system. Since thickness of the bond coating affects the life of the thermal barrier system, the uniformity of the bond coating thickness is probably also very important because a thin spot could lead to a local, premature failure. The furnace test data in table VI suggest that the bond coating should be at least 0.015 to 0.020 cm thick in order to obtain long life. The bond coating thickness values reported in table VI represent the minimum and the maximum thicknesses encountered during measurements of 2.5x1.3x0.25 cm specimens. The thicknesses on such specimens were normally measured at 11 locations. It is also likely that there might be places on the specimens that have thinner bond coating than the minimum thickness values reported in table VI since only a limited number of measurements were taken with a micrometer having a 0.65 cm diameter shaft. In fact close examination of the photomicrographs seems to confirm that such is the case. In view of this, it should be remembered that it still might be possible to use a bond coating thickness of less than 0.015 to 0.020 cm (for example, about 0.010 to 0.015 cm) provided that the bond coating is more uniformly applied so that no very thin areas are present.

The microstructures of the bond coatings before testing were very similar to the microstructures of the bond coating in figure 6, except for the thickness. The photomicrograph in figure 10a shows that the 0.003 to 0.007 cm thick bond coating was nearly completely oxidized when the NiCrAl-0.32Y/ZrO2-7.8Y2O3 thermal barrier system failed after 1419 1-hour cycles at 990° C. Increasing the bond coating thickness to between about 0.012 to 0.018 cm resulted in an increase in life to 1610 1-hour cycles and no failure (Table VI) but the bond coating was just about completely oxidized, thus, indicationg that failure was imminent. Results from the tests performed at  $1095^{\circ}$  C showed a similar trend to the one observed at  $990^{\circ}$  C. The bond coating that was 0.005 to 0.008 cm thick was completely oxidized after 46 1-hour cycles at 10950 C (Fig. 10b). Increasing the thickness to between 0.013 to 0.019 cm, the life increased to 104 1-hour cycles. The microstructures of the thicker bond coatings in the Ni-16.4Cr-5.8Al-0.32Y/ZrO<sub>2</sub>-7.8Y<sub>2</sub>O<sub>3</sub> thermal barrier system after testing at  $1095^{\circ}$  C were very similar to those in figures 8b and 9b in terms of the amount of degradation. The amount of degradation of the bond coating seems to decrease with increasing bond coating thickness as would be expected for any metallic coating. That part of the bond coating layer near the substrate seems to be only slightly degraded with bond coating thickness, and also the width of the substrate depletion zone seems to decrease with increased bond coat thickness.

The effect of the bond coating thickness seems to be independent of the bond and thermal barrier oxide coating compositions. The data in table VI suggest that the effect of Ni-16.8Cr-5.8Al-0.62Y bond coating thickness on the performance life of the Ni-16.8Cr-5.8Al-0.62Y/Zr02-7.8Y203 thermal barrier system at  $990^{\rm O}$  C was very similar to that observed with the Ni-16.4Cr-5.8Al-0.32Y/Zr02-7.8Y203 thermal barrier system. Furthermore, the data in table VI show that the bond coating thickness effect trends were very similar in the Ni-16.4Cr-5.8Al-0.32Y/Zr02-7.8Y203 and Ni-16.8Cr-5.8Al-0.32Y/Zr02-11.8Y203 thermal barrier systems.

The effect of bond coating is even more strikingly shown by the data presented in table VII. The data in table VII show that a 0.020 cm thick Ni-25.7Cr-5.6Al-0.32Y bond coating when coupled with 7.8Y2O3- and 6.1Y2O3-stabilized zirconia withstood between 635 and 656 and between 668 and 681 1-hour cycles at 1095° C, respectively. Comparing the data in tables V and VII for the Ni-25.7Cr-5.6Al-0.32Y/ $Zr0_2$ -7.8 $Y_2$ 03 thermal barrier system, it is apparent that increasing the bond coating from about 0.011 to about 0.020 cm resulted in about 2.5 fold increase in the life of the thermal barrier system. Again, it should be remembered that the bond coating thicknesses were probably not uniform and the bond coatings were thinner in many places on the specimens than the thickness values reported in tables V and VII. It is believed that it is the thin bond coating areas that limit the life of such thermal barrier systems. The data in table VII further show that the Ni-25.7Cr-5.6Al-0.32Y/Zr02-6.1 $Y_2$ 03 system has a slightly longer life than the Ni-25.7Cr-5.6Al-0.32Y/ZrO<sub>2</sub>-7.8Y<sub>2</sub>O<sub>3 system</sub>. This agrees with the data previously reported in reference 11. Furthermore, the data in table VII show very good reproductibility in the life for duplicate tested

specimens. The values reported in table VII are for individual specimens while in all other tables the values are averages of two specimens tested under identical conditions. It was observed in the data reported in tables II, III, IV, V an VI that the reproducibility of life between two specimens coated with the same thermal barrier system and tested under the same conditions did not vary by more than plus or minus 10 percent at  $1095^{\circ}$  C and less at  $990^{\circ}$  C.

It is still true that for yttrium containing bond coatings the failures of the thermal barrier systems occur within the oxide layer near the bond coating-oxide layer interface (Fig. 10). It seems that the bond coating thickness does not affect the failure mechanism. It was observed, that for bond coatings that were completely oxidized, cracks seem to appear within the bond coating (Fig. 10b). However, it is possible that the cracks within the bond coating could have been caused during cutting and polishing of the specimens for metallographic examination. Therfore, additional evaluation must be done in order to reach a more definitive conclusion as to why and how these cracks within totally oxidized bond coatings occur.

## CONCLUSIONS

A study was conducted using cyclic furnace tests to evaluate the effects of chromium, aluminum, and yttrium concentrations in the bond coating, as well as the effect of the thickness of the bond coating on the performance of the thermal barrier coating system. The data obtained in this study suggest that:

- 1. The chemical properties of the bond coating, particularly the resistance to degradation through oxidation has a very significant influence on the adherence and the performance of the thermal barrier system.
- a. The presence of yttrium is very critical. Without yttrium in the bond coating, the thermal barrier systems fail very rapidly. Yttrium in the bond coating affects the oxidation degradation of the bond coating and the failure mechanism of the thermal barrier coating. With no yttrium, the failure occurs at the substrate-bond coating interface. With a low level of yttrium in the bond coating, the failure occurs within the oxide coating near the bond coating-oxide coating interface.
- b. The concentrations of yttrium, aluminum, and/or chromium in the bond coating critically affect performance of the thermal barrier system. It was found in this study that the best bond coating was Ni-25.7Cr-5.6Al-0.32Y followed closely by Ni-19.8Cr-19.2Al-0.33Y, and Ni-19.3Al-0.52Y. These bond coatings have improved the life of the thermal barrier coating by about two times over the Ni-16.4Cr-5.1Al-0.15Y bond coating previously reported in the literature.
- 2. There is a minimum bond coating thickness of 0.010 to 0.012 cm above which any increase in thickness only slightly improves the life of the thermal barrier system. However, to assure that this thickness is achieved over the entire surface of the specimen, increasing average bond coating thickness to 0.020 cm is desirable. Significant decreases in the bond coating thickness below

this value, result in significantly decreased life of the thermal barrier system. The life of the thermal barrier system can be improved at least two times when the thickness of the bond coating is above the minimum thickness value.

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TABLE I. - TYPICAL COMPOSITIONS OF SPRAY POWDERS

AND SUPERALLOY SUBSTRATES<sup>a</sup>

Element	Composition, wt %				
	NiCrAlY	ZrO <sub>2</sub> -Y <sub>2</sub> O <sub>3</sub>	B-1900 + Hf	MAR-M-200 + Hf	
Al	b0.04 - 20.0	0.009	6.15	5.21	
В	.002	<.001	.003	.017	
С	.008	$^{ m c}_{ m ND}$	.008	.15	
Ca	<.001	.057	<.001	ND	
Co	.035	<.001	10.43	10.12	
Cr	b <sub>0</sub> .09 - 25	<.001	8.24	8.49	
Cu	.018	ND	.002	.045	
Fe	.075	.038	.06	.37	
Hf	<.01	1.60	1.22	2.08	
K	<.001	<.001	<.001	<.001	
Li	<.001	<.001	<.001	<.001	
Mg	<.001	.020	<.001	<.001	
Mn	.009	<.001	<.001	.01	
Mo	<.005	<.005	5.79	<.005	
Na	<.001	<.001	<.001	· <.001	
Nb	.005	<.005	<.005	1.22	
Ni	Major	<.001	Major	Major	
$O_2$	.019	ND	ND	ND	
P	.002	ND	ND	ND	
Pb	ND	ND	.005	.005	
S	.002	ND	.001	.01	
Si	.062	.044	.01	.19	
Sr	<.01	<.01	<.01	ND	
Та	<.05	<.02	4.13	.05	
Ti	.005	.02	ND	2.02	
v	.028	<.001	.001	<.001	
W	.<. 01	<.005	.01	11.72	
Y	<sup>6</sup> 0 - 1.6	d <sub>4.8</sub> - 19.2	<.001	ND	
Zn	<. 05	<.005	<.001	<.005	
Zr	.015	Major	.005	.08	

<sup>&</sup>lt;sup>a</sup>Impurity values reported in the table are the maximum values encountered.

<sup>&</sup>lt;sup>b</sup>Aluminum, chromium, and yttrium concentrations for various bond coatings used are given in tables preseting experimental data.

<sup>&</sup>lt;sup>c</sup>ND, not determined.

<sup>&</sup>lt;sup>d</sup>Yttria concentrations for various yttria-stabilized zirconias used are given in tables presenting experimental data.

TABLE II. - THE EFFECTS OF VARIOUS YTTRIUM-FREE BOND COATINGS ON
THE PERFORMANCE OF THE YTTRIA-STABILIZED ZIRCONIA THERMAL

# BARRIER COATINGS. CYCLIC FURNACE TESTS<sup>a</sup>

Bond coating		Thermal barrier coating		Average number of		
Composition, Thickne		Composition, Thickness, d cm		cycles to failure for two specimens		
		9900 - 2800 (				
No bond coating Ni-19.8Al -20.2Cr -16.2Cr-5.5Al  No bond coating Ni-19.8Al -20.2Cr -16.2Cr-5.5Al	0.009 .010 .011  0.010 .011	ZrO <sub>2</sub> -7.8Y <sub>2</sub> O <sub>3</sub> -11.5Y <sub>2</sub> O <sub>3</sub> -11.5Y <sub>2</sub> O <sub>3</sub> -11.5Y <sub>2</sub> O <sub>3</sub> -11.5Y <sub>2</sub> O <sub>3</sub>	.034 .036 0.044 .034 .039	b <sub>85</sub> 103 205 455 b <sub>55</sub> 68 118 326		
1095° - 280° C						
Ni-20.2Cr -16.2Cr-5.5Al Ni-20.2Cr -16.2Cr-5.5Al	0.012 .011 0.011 .010	$ZrO_2$ -7.8 $Y_2O_3$ -7.8 $Y_2O_3$ $ZrO_2$ -11.5 $Y_2O_3$ -11.5 $Y_2O_3$		27 54 13 38		

<sup>a</sup>Cycle consisted of 6 minutes heat up, 60 minutes at temperature, and 60 minutes cooling to about 280° C. Tests stopped whenever a visible crack occurred. Specimens inspected almost every 12 cycles.

bSubstrate material was B-1900 with 1.2 wt % Hf. Substrate material in all other tests was MAR-M-200 with 2.1 wt % Hf. Specimens were about 2.5×1.3×0.25 cm. A minimum of two specimens were run per each test.

<sup>&</sup>lt;sup>c</sup>Bond coating on each specimen could vary by ±0.004 cm.

dThermal barrier oxide coating on each specimen could vary by ±0.005 cm.

TABLE III. - EFFECT OF YTTRIUM CONCENTRATION IN Ni-Cr BOND COATINGS
ON YTTRIA-STABILIZED ZIRCONIA THERMAL BARRIER COATING LIFE.

CYCLIC FURNACE TEST RESULTS<sup>a</sup>

Bond coat	ing	Thermal barri	Average number of				
Composition, Thickness, d cm		Composition, Thickness, e cm		cycles to failure for two specimens <sup>b</sup>			
		990° - 280° C					
Ni-20.2Cr	0.010	ZrO <sub>2</sub> -7.8Y <sub>2</sub> O <sub>3</sub>	0.034	205			
-19.5Cr-1.53Y	.009	-7.8Y <sub>2</sub> O <sub>3</sub>	.042	300			
-19.8Cr-0.53Y	.011	-7.8Y <sub>2</sub> O <sub>3</sub>	.040	<sup>c</sup> 1550			
Ni-20.2Cr	0.011	ZrO <sub>2</sub> -11.5Y <sub>2</sub> O <sub>3</sub>	0.039	118			
-19.5Cr-1.53Y	.011	-11.5Y <sub>2</sub> O <sub>3</sub>	.040	169			
-19.8Cr-0.53Y	.012	-11.5Y <sub>2</sub> O <sub>3</sub>	.039	1537			
Ni-20.2Cr	0.010	ZrO <sub>2</sub> -17.4Y <sub>2</sub> O <sub>3</sub>	0.037	90			
-19.5Cr-1.53Y	.009	-17.4Y <sub>2</sub> O <sub>3</sub>	.034	152			
-19.8Cr-0.53Y	.013	-17.4Y <sub>2</sub> O <sub>3</sub>	.040	612			
Ni-20.2Cr	0.011	ZrO <sub>2</sub> -24.4Y <sub>2</sub> O <sub>3</sub>	0.042	75			
-19.5Cr-1.53Y	.011	-24.4Y <sub>2</sub> O <sub>3</sub>	.046	93			
-19.8Cr-0.53Y	.011	-24.4Y <sub>2</sub> O <sub>3</sub>	.039	354			
	1095° - 280° C						
Ni-20.2Cr	0.012	ZrO <sub>2</sub> -7.8Y <sub>2</sub> O <sub>3</sub>	0.043	27			
-19.5Cr-1.53Y	.010	-7.8Y <sub>2</sub> O <sub>3</sub>	.043	47			
-19-8Cr-0.53Y	.010	-7.8Y <sub>2</sub> O <sub>3</sub>	.039	56			
Ni-20.2Cr	0.011	ZrO <sub>2</sub> -11.5Y <sub>2</sub> O <sub>3</sub>	0.043	13			
-19.5Cr-1.53Y	.011	-11.5Y <sub>2</sub> O <sub>3</sub>	.039	24			
-19.8Cr-0.53Y	.011	-11.5Y <sub>2</sub> O <sub>3</sub>	.044	46			
Ni-20.2Cr	0.012	ZrO <sub>2</sub> -17.4Y <sub>2</sub> O <sub>3</sub>	0.043	10			
-19.5Cr-1.53Y	.009	-17.4Y <sub>2</sub> O <sub>3</sub>	.046	20			

 $^{2}$ MAR-M-200 alloy with 2.1 wt % Hf was used as a substrate material; Specimens were about 2.5×1.3-0.25 cm. A minimum of two specimens were run per each test.

<sup>b</sup>Cycle consisted of 6 minutes heat up, 60 minutes at temperature, 60 minutes cooling to about 280° C. Test stopped whenever a visible crack occurred.

<sup>&</sup>lt;sup>c</sup>No failure.

dBond coating on each specimen could vary by ±0.004 cm.

<sup>&</sup>lt;sup>e</sup>Thermal barrier oxide coating on each specimen could vary by ±0.005 cm.

TABLE IV. - EFFECT OF YTTRIUM CONCENTRATION IN NI-Al BOND COATINGS
ON YTTRIA-STABILIZED ZIRCONIA THERMAL BARRIER COATING LIFE.

# CYCLIC FURNACE TEST RESULTS<sup>a</sup>

Bond coating		Thermal barrier coating		Average number of			
Composition, Thickness, d cm		Composition, wt %	Thickness, e	cycles to failure for two specimens <sup>b</sup>			
		990° – 280° C					
Ni-19.8Al	0.009	ZrO <sub>2</sub> -7.8Y <sub>2</sub> O <sub>3</sub>	0.042	103			
-19.4Al-1.60Y	.012	-7.8Y <sub>2</sub> O <sub>3</sub>	.041	233			
-19.3Al-0.52Y	.010	-7.8Y <sub>2</sub> O <sub>3</sub>	.042	<sup>c</sup> 1389			
Ni-19.8Al	0.010	ZrO <sub>2</sub> -11.5Y <sub>2</sub> O <sub>3</sub>	0.034	68			
-19.4Al-1.60Y	.011	-11.5Y <sub>2</sub> O <sub>3</sub>	.040	150			
-19.3Al-0.52Y	.012	-11.5Y <sub>2</sub> O <sub>3</sub>	.042	<sup>C</sup> 1256			
Ni-19.8Al	0.010	ZrO <sub>2</sub> -17. 4Y <sub>2</sub> O <sub>3</sub>	0.040	54			
-19.4Al-1.60Y	.010	-17. 4Y <sub>2</sub> O <sub>3</sub>	.050	85			
-19.3Al-0.52Y	.011	-17. 4Y <sub>2</sub> O <sub>3</sub>	.044	448			
Ni-19.8Al	0.010	ZrO <sub>2</sub> -24. 4Y <sub>2</sub> O <sub>3</sub>	0.042	33			
-19.4Al-1.60Y	.011	-24. 4Y <sub>2</sub> O <sub>3</sub>	.043	59			
-19.3Al-0.52Y	.011	-24. 4Y <sub>2</sub> O <sub>3</sub>	.044	259			
	1095° - 280° C						
Ni-19.4Al-1.60Y	0.010	ZrO <sub>2</sub> -7.8Y <sub>2</sub> O <sub>3</sub>	0.038	70			
-19.3Al-0.52Y	.011	-7.8Y <sub>2</sub> O <sub>3</sub>	.038	198			
Ni-19.4Al-1.60Y	0.010	ZrO <sub>2</sub> -11.5Y <sub>2</sub> O <sub>3</sub>	0.040	35			
-19.3Al-0.52Y	.011	-11.5Y <sub>2</sub> O <sub>3</sub>	.042	160			
Ni-19.4Al-1.60Y	0.012	ZrO <sub>2</sub> -17.4Y <sub>2</sub> O <sub>3</sub>	0.043	29			
Ni-19.4Al-1.60Y	0.010	ZrO <sub>2</sub> -24.4Y <sub>2</sub> O <sub>3</sub>	0.040	19			

 $<sup>^3</sup>$ MAR-M-200 alloy with 2.1 wt % Hf was used as a substrate material; Specimens were about 2.5×1.3×0.25 cm. A minimum of two specimens were run per each test.

bCycle consisted of 6 minutes heat up, 60 minutes at temperature, 60 minutes cooling to about 280° C. Test stopped whenever a visible crack occurred.

<sup>&</sup>lt;sup>c</sup>No failure.

dBond coating on each specimen could vary by ±0.004 cm.

eThermal barrier oxide coating on each specimen could vary by ±0.005 cm.

TABLE V. - EFFECTS OF CHROMIUM AND ALUMINUM CONCENTRATIONS IN Ni-Cr-Al-Y BOND COATINGS ON YTTRIA-STABILIZED THERMAL BARRIER

COATING LIFE. CYCLIC FURNACE TEST RESULTS<sup>a</sup>

$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Bond coating		Thermal barrier coating		Average number of	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	<u> </u>	•	_		cycles to failure for two specimens <sup>b</sup>	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		9	90° – 280° C			
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	-16.4Cr-5.8Al-0.32Y -19.8Cr-19.2Al-0.33Y -16.6Cr-10.6Al-0.33Y Ni-25.7Cr-5.6Al-0.32Y -19.8Cr-19.2Al-0.33Y -16.6Cr-10.6Al-0.33Y	.009 .011 .013 0.010 .014 .010	-7.8Y <sub>2</sub> O <sub>3</sub> -7.8Y <sub>2</sub> O <sub>3</sub> -7.8Y <sub>2</sub> O <sub>3</sub> -7.8Y <sub>2</sub> O <sub>3</sub> -11.8Y <sub>2</sub> O <sub>3</sub> -11.8Y <sub>2</sub> O <sub>3</sub>	.044 .036 .034 0.032 .033 .039	<sup>C</sup> 1500 <sup>C</sup> 1500 <sup>C</sup> 1500 <sup>C</sup> 1500 <sup>C</sup> 1500	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$					·	
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$		10	95° - 280° C			
INTELY OUTERS AND AND AND A CONTRACT OF A CONTRACT AND	-16.4Cr-5.8Al-0.32Y -19.8Cr-19.2Al-0.33Y -16.6Cr-10.6Al-0.33Y Ni-25.7Cr-5.6Al-0.32Y -19.8Cr-19.2Al-0.33Y	.012 .012 .011 0.011 .010	-7.8Y <sub>2</sub> O <sub>3</sub> -7.8Y <sub>2</sub> O <sub>3</sub> -7.8Y <sub>2</sub> O <sub>3</sub> -7.8Y <sub>2</sub> O <sub>3</sub> ZrO <sub>2</sub> -11.8Y <sub>2</sub> O <sub>3</sub> -11.8Y <sub>2</sub> O <sub>3</sub>	.036 .032 .031 0.034 .035 .031	114 208 152 165 98	

 $<sup>^{</sup>a}B$ -1900 alloy with 1.2 wt % Hf was used as a substrate material; Specimens were about 2.5×1.3×0.25 cm. A minimum of two specimens were run per each test.

<sup>&</sup>lt;sup>b</sup>Cycle consisted of 6 minutes heat up, 60 minutes at temperature, 60 minutes cooling to about 280° C. Test stopped whenever a crack occurred. Values are averages of the two specimens.

c<sub>No</sub> failure.

dBond coating on each specimen could vary by ±0.004 cm.

eThermal barrier oxide coating on each specimen could vary by ±0.005 cm.

TABLE VI. - EFFECT OF BOND COATING THICKNESS ON YTTRIA-STABILIZED

ZIRCONIA THERMAL BARRIER COATING LIFE. CYCLIC FURNACE

TEST RESULTS<sup>a</sup>

Bond coating		Thermal barrier coating		Average number of			
Composition, wt %	Thickness range, cm	Composition, wt %	Thickness, d	cycles to failure for two specimens <sup>b</sup>			
		9900 - 2800 C					
-0.32Y -0.32Y -0.32Y -0.32Y Ni-16.4Cr-5.8Al-0.32Y -0.32Y -0.32Y	.012018 .018026 .026032 0.002-0.007 .004010 .009015 .018027 .020027 0.005-0.009 .007010	ZrO <sub>2</sub> -7.8Y <sub>2</sub> O <sub>3</sub> -11.8Y <sub>2</sub> O <sub>3</sub> -7.8Y <sub>2</sub> O <sub>3</sub>	0.028 .028 .032 .032 .030 0.036 .034 .039 .038 .039	1419 1529 <sup>c</sup> 1610 <sup>c</sup> 1610 <sup>c</sup> 1629 696 780 1294 1119 1132 676 726 877			
	1095° - 280° C						
Ni-16.4Cr-5.8Al-0.32Y -0.32Y -0.32Y Ni-16.4Cr-5.8Al-0.32Y -0.32Y -0.32Y	0.005-0.008 .007010 .013019 0.004-0.007 .007010	ZrO <sub>2</sub> -7.8Y <sub>2</sub> O <sub>3</sub> -7.8Y <sub>2</sub> O <sub>3</sub> -7.8Y <sub>2</sub> O <sub>3</sub>	0.033 .036 .038 0.033 .036 .038	46 60 104 23 35 60			

 $<sup>^{2}</sup>$ B-1900 alloy with 1.2 wt % Hf was used as the substrate material; Samples were about 2.5×1.3×0.25 cm. A minimum of two samples were run at each test.

<sup>&</sup>lt;sup>b</sup>Cycle consisted of 6 minutes heat up, 60 minutes at temperature, 60 minutes cooling to about 280° C, Testing stopped whenever a crack occurred.

cNo failure.

dThermal barrier oxide coating on each specimen could vary by  $\pm 0.005$  cm.

# TABLE VII. - EFFECT OF Ni-25.7Cr-5.6Al-0.32Y BOND COATING THICK-NESS ON TWO YTTRIA-STABILIZED ZIRCONIA THERMAL BARRIER COATINGS AT 1095° C. CYCLIC FURNACE TEST RESULTS<sup>2</sup>

Bond coating	Thermal barri	Cycles to		
Composition, wt $\%$	Maximum thickness, cm	Composition, wt %	Maximum thickness, cm	failure <sup>b</sup>
Ni-25.7Cr-5.6Al-0.32Y	0.020	ZrO <sub>2</sub> -6.1Y <sub>2</sub> O <sub>3</sub>	0.041	668
-0.32Y	.021	-6.1Y <sub>2</sub> O <sub>3</sub>		68 <b>1</b>
Ni-25.7Cr-5.6Al-0.32Y	0.020	ZrO <sub>2</sub> -7.8Y <sub>2</sub> O <sub>3</sub> -7.8Y <sub>2</sub> O <sub>3</sub>	0.042	656
-0.32Y	.020		.042	635

<sup>a</sup>B-1900 alloy with 1.2 wt % Hf was used as the substrate material; Samples were 2.5×1.3×0.25 cm. One sample was run per each test.

<sup>b</sup>Cycle consisted of 6 minutes heat up, 60 minutes at temperature, and 60 minutes cooling to about 280° C. Testing stopped whenever a crack occurred.

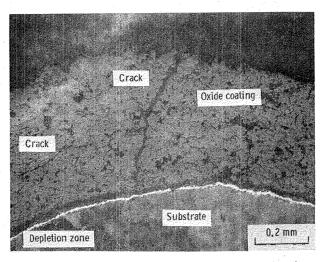
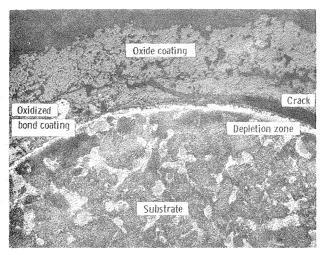


Figure 1. - Light optical photomicrograph of edge of B-1900 + Hf specimen coated with  $ZrO_2$ -7.8 $Y_2O_3$  after testing for 85 cycles at 990 $^{\rm O}$  C. (Cycle, 6 minutes heat up, 60 minutes at temperature, 60 minutes cooling to about 280 $^{\rm O}$  C.)



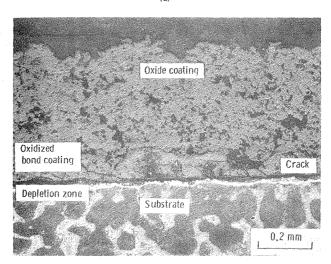


Figure 2. - Light optical photomicrograph of: (a) the edge of MAR-M-200 + Hf specimen coated with Ni-20.2Cr/ZrO<sub>2</sub>-7.8Y $_2$ O $_3$  after 200 1-hour cycles at 990 $^0$  C; (b) the flat surface of MAR-M-200 + Hf specimen coated with Ni-19.8AI/ZrO $_2$ -7.8Y $_2$ O $_3$  after 103 1-hour cycles at 990 $^0$  C. (Cycle, 6 minutes heat up, 60 minutes at temperature, and 60 minutes cooling to about 280 $^0$  C.)

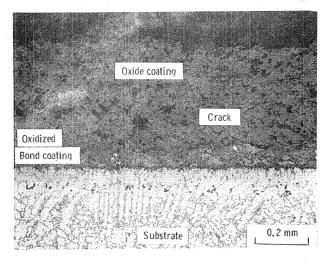
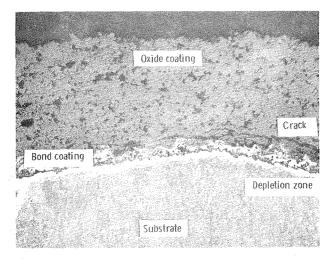


Figure 3. - Light optical photomicrograph of flat surface of MAR-M-200 + Hf specimen coated with Ni-19.8Cr-0.53Y and  $ZrO_2$ -7.8Y $_2O_3$  after 1550 1-hour cycles at  $990^{\circ}$  C and no failure. (Cycle, 6 minutes heat up, 60 minutes at temperature, and 60 minutes cooling to about  $280^{\circ}$  C.)



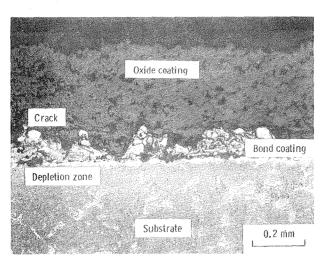


Figure 4. - Light optical photomicrograph of: (a) the edge surface of MARM-200 + Hf specimen coated with Ni-19.8Cr-0.53Y and ZrO $_2$ -7.8Y $_2$ O $_3$  after 56 1-hour cycles at 1095 $^{\rm O}$  C, and (b) the flat surface of MAR-M-200 + Hf specimen coated with Ni-19.3AI-0.52Y and ZrO $_2$ -7.8Y $_2$ O $_3$  after 198 1-hour cycles at 1095 $^{\rm O}$  C. (Cycle, 6 minutes heat up, 60 minutes at temperature, and 60 minutes cooling to about 280 $^{\rm O}$  C.)

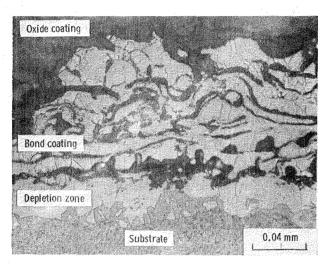


Figure 5. - Light optical photomicrograph of flat surface of MAR-M-200 + Hf specimen coated with Ni-19.3Al-0.52Y and  $\rm ZrO_2$ -7.8Y $_2\rm O_3$  after 198 1-hour cycles at 1095 $^0$  C. (Cycle, 6 minutes heat up, 60 minutes at temperature, and 60 minutes cooling to about 280 $^0$  C.)

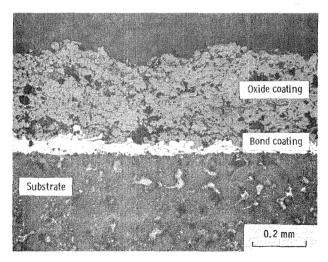
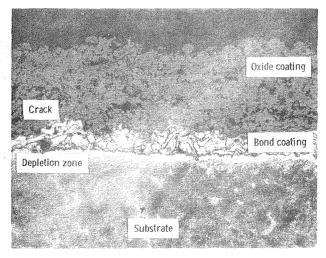


Figure 6. - Light optical photomicrograph of flat surface of B-1900 + Hf specimen coated with Ni-25.7Cr-5.6Al-0.32Y and ZrO<sub>2</sub>-7.8Y<sub>2</sub>O<sub>3</sub> after plasma spraying and no cyclic testing,



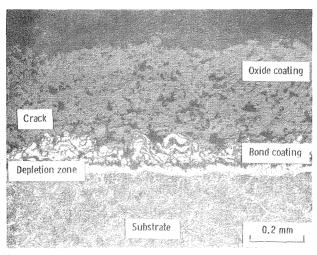
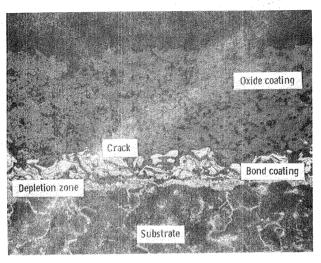


Figure 7. – Light optical photomicrograph of B-1900 + Hf specimen coated with Ni-25, 7Cr-5, 6Al-0, 32Y and ZrO<sub>2</sub>-7, 8Y $_2$ O $_3$ : (a) flat surface of specimen after 1500 1-hour cycles and no failure at 990 $^{\rm o}$  C, and (b) flat surface of specimen after 249 1-hour cycles and failure at 1095 $^{\rm o}$  C. (Cycle, 6 minutes heat up, 60 minutes at temperature, and 60 minutes cooling to about 280 $^{\rm o}$  C.)



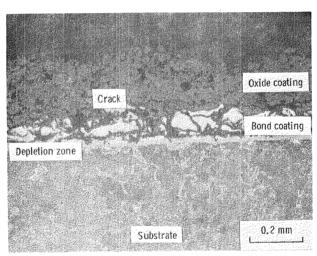
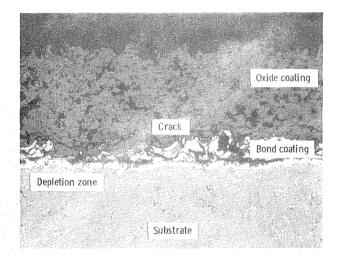


Figure 8. – Light optical photomicrograph of B–1900 + Hf specimen coated with Ni–19.9Cr–19.2Al–0.33Y and ZrO $_2$ –7.8Y $_2$ O $_3$ : (a) flat surface of specimen after 1500 1-hour cycles and no failure at 990 $^{\rm O}$ C, and (b) flat surface of specimen after 190 1-hour cycles and fialure at 1095 $^{\rm O}$ C. (Cycle, 6 minutes heat up, 60 minutes at temperature, and 60 minutes cooling to about 280 $^{\rm O}$ C.)



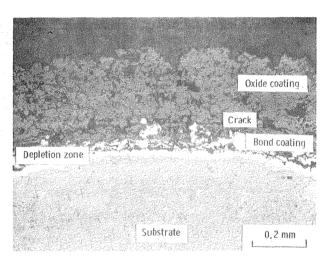
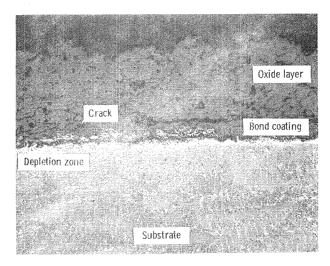


Figure 9. - Light optical photomicrographs of B-1900 + Hf specimens coated with Ni-16.6Cr-10.6Al-0.33Y and ZrO<sub>2</sub>-7.8Y<sub>2</sub>O<sub>3</sub>: (a) flat surface of specimen after 1500 1-hour cycles at  $990^{\circ}$  C and no failure, and (b) edge of specimen after 150 1-hour cycles at  $1095^{\circ}$  C and failure. (Cycle, 6 minutes heat up, 60 minutes at temperature, and 60 minutes cooling to about  $280^{\circ}$  C.)



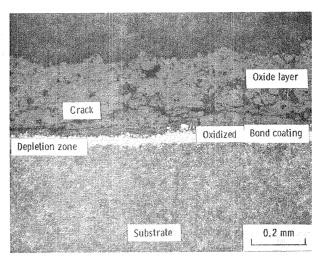


Figure 10. – Light optical photomicrographs of flat surfaces of B-1900 + Hf specimens coated with : (a) 0.003 to 0.007 cm Ni-16.4Cr-5.8Al-0.32Y and Zr0<sub>2</sub>-7.8Y $_2$ 0 $_3$  after 1419 1-hour cycles at 990 $^0$  C and failure, and (b) 0.005 to 0.008 cm Ni-16.4Cr-5.8Al-0.32Y and Zr0 $_2$ -7.8Y $_2$ 0 $_3$  after 46 1-hour cycles at 1095 $^0$  C and failure. (Cycle, 6 minutes heat up, 60 minutes at temperature, and 60 minutes cooling to about 280 $^0$  C.)

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